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Mazdoor Kisan Shakti Sangathan

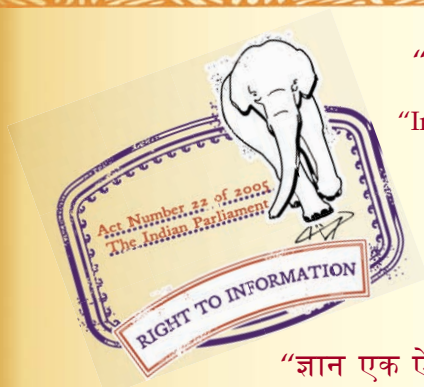
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“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 12026-1 (1986): Guide for the determination of the aromatic hydrocarbon content of new insulating oils, Part 1: Asorption chromatographic method [ETD 3: Fluids for Electrotechnical Applications]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

GUIDE FOR THE
DETERMINATION OF THE AROMATIC
HYDROCARBON CONTENT OF NEW
INSULATING OILS

PART 1 ADSORPTION CHROMATOGRAPHIC METHOD

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NEW DELHI 110002

Indian Standard

GUIDE FOR THE DETERMINATION OF THE AROMATIC HYDROCARBON CONTENT OF NEW INSULATING OILS

PART 1 ADSORPTION CHROMATOGRAPHIC METHOD

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Indian Standard

GUIDE FOR THE DETERMINATION OF THE AROMATIC HYDROCARBON CONTENT OF NEW INSULATING OILS

PART 1 ADSORPTION CHROMATOGRAPHIC METHOD

0. FOREWORD

0.1 This Indian Standard (Part 1) was adopted by the Indian Standards Institution on 24 October 1986, after the draft finalized by the Liquid and Gaseous Dielectrics Sectional Committee had been approved by the Electrotechnical Division Council.

0.2 This standard is being brought out as a guide for determination of the aromatic hydrocarbon content of new insulating oils.

0.3 This standard is being brought out in two parts. Part 2 covers the infrared spectrophotometric method of determination of aromatic hydrocarbon content in new insulating oils.

0.4 In the preparation of this standard, assistance has been derived from IEC Pub 590 (1977) 'Determination of the aromatic hydrocarbon content of new mineral insulation oils', issued by the International Electrotechnical Commission.

0.5 Determination of olefinic plus aromatic hydrocarbon for petroleum and its products that are substantially free of butenes and that have 90 percent distillation not over 315°C, and determination of aromatics in the aviation gasolines, etc, by gas chromatography has been covered in IS : 1448 [P : 104]-1981* and IS : 1448 [P : 48]-1978†.

0.5.1 This method describes the measurement of the percentage by mass of polar compounds and compound containing at least one aromatic ring per molecule present in low molecular mass insulating oils using analytical grades of *n*-pentane and ethoxyethane as solvents.

*Methods of test for petroleum and its products, [P : 104] 'Aromatics in light naphthas and aviation gasolines by gas chromatography.

†Methods of test for petroleum and its products, [P : 48] Olefines and aromatic content (*first revision*).

0.6 New insulating oils are covered in IS : 335-1983*.

0.7 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard (Part 1) describes the method of determination of the aromatic hydrocarbon content of new insulating oils by adsorption chromatographic method.

1.2 Adsorption chromatographic test method describes the measurement of the percentage by mass of polar compounds and compounds containing at least one aromatic ring per molecule present in low molecular mass insulating oils.

2. TERMINOLOGY

2.1 For the purpose of this standard, the relevant definitions given in IS : 335-1983‡, shall apply.

3. APPARATUS

3.1 Adsorption Column (Fig. 1) — It consists of a glass tube 50 cm long, having 1 cm internal diameter, drawn out into a taper at one end (internal diameter of the tapered end : 2 mm) and having a bulb of about 50 ml capacity at the upper end.

3.1.1 This column is calibrated with one mark corresponding to the volume of 10 g of silica gel.

3.2 Vacuum Pump — It is fitted with a cold trap.

3.2.1 Graduated measuring cylinders (1 division = 1 ml) of 25 ml capacity.

3.2.2 Heating Device — Any device (water or oil bath, heating mantle) capable of being maintained at any temperature in the range of 50 to 100°C.

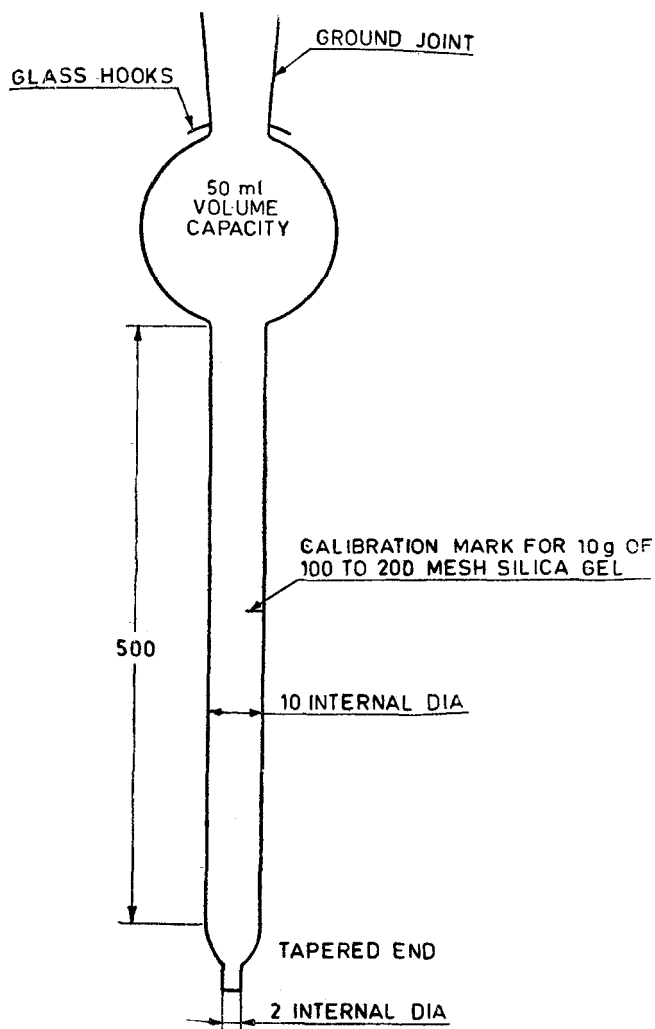
3.2.3 Evaporation Flasks

3.2.4 Weighing Bottle (Volume Approximately 20 ml)

*Specification for new insulating oils (*third revision*).

†Rules for rounding off numerical values (*revised*).

‡Specification for new insulating oils (*third revision*).



All dimensions in millimetres.

FIG. 1 GLASS ADSORPTION COLUMN

3.3 Reagents

3.3.1 Chromatographic grade silica gel 100 to 200 mesh is activated in an oven at 160°C for 8 h just before use. The preferred range of pore size will be 2 Nm to 4 Nm.

NOTE — It may be noted that silica gel from different sources and from different batches have wide variations of adsorption (chromatographic) properties.

3.3.2 *n*-pentane, analytical grade.

3.3.3 Ethoxyethane, analytical grade.

4. TEST METHOD

4.0 Adsorption Chromatography Analysis

4.0.1 The sample diluted in *n*-pentane is separated on a column filled with activated silica gel.

Two fractions are separated:

- a) Saturated hydrocarbons (paraffins and naphthenes); and
- b) Polar compounds (aromatic hydrocarbons plus sulphur, oxygen or nitrogen compounds).

4.1 Insert a plug of cotton wool in the lower end of the clean dry glass adsorption column to retain the adsorbent.

4.2 Immediately after being activated, and without being allowed to cool, the silica gel is poured into the column which is tapped gently to compact the silica gel uniformly until the level remains at the 10 g mark.

4.3 Place a small plug of cotton wool on top of the gel. As far as possible, activated silica gel must be kept away from the moisture of the air (for example, it is recommended to seal the column during the packing process and the cooling of the adsorbent).

4.4 Weigh approximately 0.5 g (to the nearest ± 0.0002 g) of oil sample into a 20-ml weighing bottle and dilute with 5 ml of *n*-pentane.

4.5 Pour 10 ml of *n*-pentane into the column containing the adsorbent. Elute this solvent and just before the level of the solvent reaches the upper face of the adsorbent and the diluted sample.

4.6 Thoroughly wash out the weighing bottle with the minimum amount of *n*-pentane. Pour this solvent into the top of the column.

4.7 When all the diluted sample has entered the silica gel, add further 10 ml quantities of *n*-pentane so that the column is never allowed to run dry.

4.8 Collect the fractions in 25 ml measuring cylinders.

4.9 After the diluted sample has been added and between 17 ml and 20 ml of n-pentane has been collected determine the cut-out point between the saturated and polar compounds (that is, aromatics) by the following procedure:

- a) Take one drop of the percolate on a Whatman No. 1 filter paper;
- b) Allow the solvent to evaporate;
- c) If the saturated hydrocarbons are completely eluted, no oil residue will be visible on the paper by transmitted light; and
- d) Repeat this every 2 ml until a drop leaves no more oil residue on the filter paper.

4.10 The fractions eluted from the introduction of the diluted sample until complete elution of saturated hydrocarbons are collected in a previously weighed evaporation flask (fraction 1).

4.11 Elute the polar compounds till adsorbed in the column with 50 ml ethoxyethane.

4.12 Collect the eluted fraction in a weighed evaporation flask (fraction 2).

4.13 The fractions 1 and 2 are separately evaporated first at 50°C and finally under vacuum (2 torr) at 100°C (1 torr = 1.3×10^2 Pa) during a sufficient time to get rid of the solvent.

NOTE — During the evaporation care should be taken not to lose light fractions of the sample itself.

4.14 Weigh the two residues to the nearest ± 0.0002 g (mass *S* for the fraction 1 and mass *A* for the fraction 2).

NOTE — The procedure has been correctly applied if the masses of the residues (*S* + *A*) is between 98 percent and 102 percent of the mass of the original sample.

4.15 Results

4.15.1 The percentage of aromatic hydrocarbons (and other polar compounds) = $\frac{A \times 100}{A + S}$

where

A = mass (g) aromatic hydrocarbons (and other polar compounds), and

S = mass (g) of saturated hydrocarbons.

NOTE — For the application of this method it may be assumed that all polar derivatives are compound hydrocarbons.

4.16 Report

4.16.1 The following criteria may be used for judging the acceptability of results (95 percent confidence):

Repeatability (r) : Duplicate results by the same operator should be considered suspect if they differ by more than the amount given in the following table

Reproducibility (R) : The results submitted by each of two laboratories should be considered suspect if the two results differ by more than the amount given in the following table

<i>Aromatic Carbon</i>	<i>Percentage Aromatic</i>	<i>Hydrocarbons</i>
Content (CA)	≥ 10	< 10
Repeatability	5	5
Reproducibility	14	20